Pro Gradu
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Creep in metals

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Tiivistelmä — Referat — Abstract

Creep is time dependent plastic malformation of solids, that happen in static stress and temperature when threshold values are met. Creep occurs at high temperature, meaning temperature more than 30% of material's absolute melting temperature (this limit is a little lower with plastics, and higher in ceramics). The malformations it causes can lead to rupture, which usually happen in a short time compared to the duration of the whole process.

The creep effect itself is known from already the 19th century, and for metals it's quite clear that diffusion is always present in creep (Coble and Nabarro-Herring creep), and that dislocations can increase the rate of creep strain.

Effects of creep can be seen e.g. in power plants and engines, where turbine blades, turbines, pipes and vessels are all the time at high temperature and stress. Also creep relaxation is often 'loosening' bolts which needs to be retightened. In regular office creep can be seen in paper clips, especially in plastic ones, which relax and lose grip fast because of the low melting point of plastics. Creep, because it usually needs long time to be visible, has been part of accidents, too, e.g. in '9/11'.

Creep appears in 3 stages (primary (transient), secondary (steady-state), tertiary), and depending on the application, either secondary or tertiary is the most important one. The secondary creep is important for displacement-, buckling- and relaxation-limited situations, and tertiary for the rupture-limited ones.

Avainsanat — Nyckelord — Keywords
Creep, dislocation, diffusion, Coble, Nabarro-Herring, Harper-Dorn

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Contents

1 Introduction

Collision of the planes didn't (alone) bring the World Trade Center towers in New York, USA, down; it needed also quite much heat (from burning kerosene) and (normal) stress from the building's top part [1]. And time. Of course the collapse needed deformation of the metal bars, forming the building frame, which can happen both elastically (target can return to original state, e.g. a spring with reasonable force) and plastically (permanently, e.g. the spring stretched too long). There were both kinds of deformations, and of the plastic deformation one was creep.

Creep can also be seen in everyday life with e.g. paper clips. At a normal room temperature paper clips made from metal keep their form with thicker pile of papers, but when heat is increased, the ability to keep papers together is lost without any extra force. Likewise one reason for the light bulb filament break can be creep.

In short, creep is the tendency of a solid material to deform permanently with time under the influence of (static) stresses. It consists of creep relaxation, creep damage and creep fracture.

Creep occurs in all solid materials, and in some way it's always present when material is heated up (over a threshold temperature) and kept at a constant pressure. Within old houses, effects of creep can be seen e.g. in lead pipes. In bigger scale, creep can be observed e.g. in glaciers (one of the ways for glacier to move is the glacier creep). From the beginning of the creep research it was clear, that classification to rigid bodies and liquids can be completely conditional, because of the way how materials, that creep, are behaving (e.g. the above mentioned glaciers).

Nowadays creep is especially important (within industrial) manufacturing parts with high temperature or for usage in it. The first is presented in chapter 3.2, and the latter in 3.1. As mentioned, creep occurs also in nature, and can even make some accidents even worse when materials are either a long time at 'high temperature' (high means 30% of absolute melting temperature in metals), or heated up when load is kept constant (the latter is presented in chapter 3.3).

2 History

Creep was researched already in the 19th century, and the first paper about it was published in 1910 [2]. In the 19th century, part of the theory of mechanical properties of the materials was already known, but it wasn't able to explain the accidents that happened when stresses were not critical. So, something else was needed for explaining these accidents. Creep, which states that deformation increases when load is constant, wasn't the only time-dependent effect that was discovered at that time; the relaxation was also discovered at the same time, which showed that when the deformation is kept constant, the stress decreases.

Creep research (and hence papers about it) can be divided into two main groups. First group focuses on specific engineering problems by utilizing the framework of the solids mechanics. This is quite logical, because machine parts, pipes and (e.g. pressure) vessels were also at that time used at high temperatures and pressures. The other group bases their studies at materials science, and tries to explain creep through e.g. microstuctures. Mere, the existing one-dimensional models require many parameters, which are hard to determinate. At first all publications about creep were in the first group, but the second group's first publication was made in 1929 [3]. It introduced the Norton law instead of Hooke's law in a one dimensional model, and was generalized to three-dimensional case in 1934 [4].

Before the World War 2, creep research was made mainly in metals, but after it also the polymeric materials were studied. It was a completely new area, because classical theories about creep didn't have any way to simulate the behaviour of plastics. Classical theories were able to describe the effects in polycrystalline structures of metals, but the polymer chains in plastics were completely different.

The equations describing different phases of creep were found in the same order as they're occurring in materials; first for primary creep (chapter 4.4.1), 1950s for secondary creep (using Norton's law) (chapter 4.4.2), and 1958 for tertiary creep (chapter 4.4.3) [5].

3 Occurrences of creep

3.1 Power plants and engines

As can be seen in chapter 4.2, temperature has quite big effect on the amount of creep. It's an especially remarkable 'problem' in heat engines (and in parts of those). Because the maximum efficiency of (ideal) heat engine is defined as

$$\eta_{\mathtt{max}} = 1 - \frac{T_c}{T_h}$$

where T_c is the absolute temperature of the cold sink (in Kelvins) and T_h that of the hot source, it can be seen that to get more efficiency, it's easier to rise the temperature than to try to cool down the cold sink. This adds quite many requirements for materials to be usable at temperatures at which e.g. a jet engine operates (1600°C [6]). This temperature is actually higher than the melting point of the material of turbine blades, but blades are cooled while used. Also cooling is quite precise; too much cooling drops the efficiency, too little lets the blades melt.

High temperatures occurs also in power plants. Most of the power plants in the world are based on steam turbines, where the high temperatures affect the steam pipes, boilers, and turbines themselves. Because of creep (especially, creep relaxation) bolts in all parts need to be tightened within certain periods to prevent leakages. Example of this can be found in chapter 4.6.1.

What is surprising, is that it seems that radiation in nuclear reactors doesn't increase the rate of creep [7]. Still, the same effects and problems with the parts, where steam is used or transferred, can be found in nuclear plant as in other heat power plants.

3.2 Manufacturing

Creep can also occur when parts/products are manufactured, not only in use. This can be coped with the same way as trying to prevent creep effect during the usage. Some ways to prevent creep can be found at chapter 6.

When building research equipment for particle physics, creep makes things quite hard. In CLIC [1], there's need to build parts by combining a stack of OFHC (Oxygen-free high conductivity) copper discs to one solid piece [8]. Discs are manufactured using μm precision, which should be present also in the final product. Discs are combined with diffusion welding, which allows making the final product seamless. On the other hand, a problem is that temperature and pressure used in diffusion welding make also creep to appear during the process and in the final product. Currently there's on-going research about the best parameters for this combination, which would produce at the same time a seamless structure and as little as possible creeping that's 'losing' the precision of initial structures [9] [10]. The temperature used in the process is around 1025°C, which is approximately 96% of absolute melting temperature of copper (1083°C) [9]. The pressures used in parameter tests were 0.05 MPa (25 kg weight on the top) and 0.1 MPa (50 kg weight on the top) [9].

The manufacturing process, where creep is problematic, is quite simple. OFHC copper discs are stacked to a horizontal stack (figure 1), aligned, and put to an oven (with hydrogen atmosphere) with predefined force added (figure 2) [8]. As can be thought from the stack's construction, the pressure towards the lower discs is much higher than on the top discs because of the weight of the copper. The structure is made in many parts, but still the lower parts are experiencing more stress than just the one added separately at the top. The reason for using a horizontal stack instead of a vertical one is to make the alignment easier and to keep it that way. In vertical stack, it's hard to keep parts in place without extra supports, which at high temperature can cause also malformations.

The situation is simulated using finite element method (FEM) [10]. Multiple deformation mechanism maps are provided for pure copper, but in this case a special one is needed for OFHC copper. In addition, some more research is needed for high temperature situations for modelling the diffusion welding process. Because of the relatively low stress (at least at the top of the pile) also studies of Harper-Dorn creep (chapter 5.4) might be needed.

[1] The Compact Linear Collider

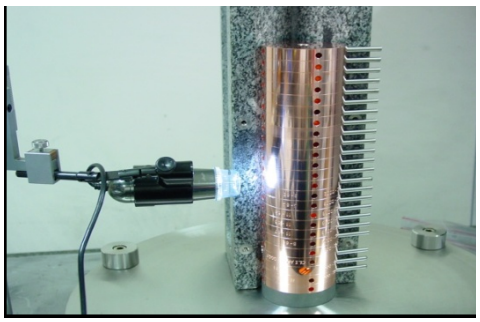

Figure 1: Building and inspecting copper disc stack before diffusion welding [8]

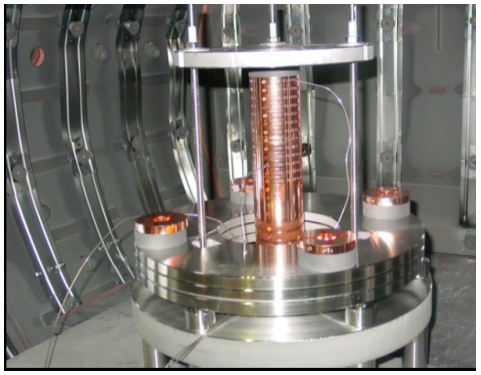

Figure 2: Copper disc stack in bonding [8]

3.3 Creep in disasters

Because the occurrence of creep damage needs time, and the damages are visible only at the tertiary stage, creep has been causing accidents and disasters. Two of events this kind are Big Dig tunnel collapse in Boston 1996 [11] (mentioned here even though it isn't related to creep in metals but to creep in general) and the WTC collapse ('9/11') in 2001.

'Big Dig' is a nickname for a 5,6 km long tunnel in Boston, which was built to reroute the Interstate 93 traffic from the city centre. The construction costs were over the budget, and the construction was plagued by e.g. leaks, design flaws and use of substandard materials. All these flaws have caused various malformations to structures, which have also led to issues. But the issue with creep is the most known.

In the 1996 accident, part of a concrete ceiling detached from the roof, and fell (with supporting structures) onto the vehicle passing by that place, causing one death, one injury, and the closure of that section of the tunnel for almost one year (figure 3). This accident was investigated by The National Transportation Safety Board (NTSB), and they released their report on the one year anniversary of the disaster [11]. According to this report, the major cause of accident was 'epoxy creep' (creep in epoxy used in adhesive anchors).

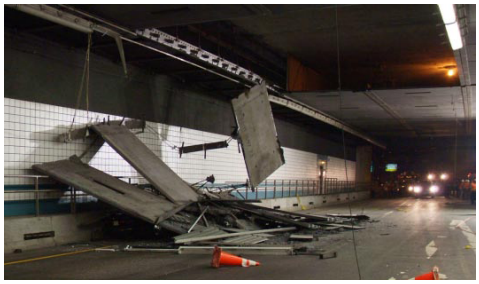

Figure 3: Post-accidental scene in tunnel. The crushed passenger car is also visible under the debris [11]

Figure 4: Other adhesive anchors photographed after the accident [11]

The tunnel ceiling (built with concrete plates) was anchored to the roof with so called adhesive anchors (in figure 5). In the anchor structure, there's a hole drilled to the concrete roof of the tunnel, filled with epoxy, which 'glued' the anchor rod to place. The roof hanger's function is just to connect different rods, allowing a one point connection to the ceiling and equalising the stress to the anchors. Because the epoxy used in some parts of the tunnel wasn't creep resistant, it started to creep (which causes malformation at tertiary stage) and allowed anchor to come out from the roof (as can be seen in figure 4). According to the report this could have been detected with inspections (even the tertiary stage is usually short, in this case the damages could have been visible long enough before the accident).

Another accident that creep had it's effect on is the collapse of the WTC towers, which was caused by two commercial airplanes with tanks full of kerosene hitting the towers. This accident was analysed by multiple articles in magazines (e.g. Why Did the World Trade Center Collapse? - Simple Analysis in Journal of Engineering mechanisms [1]) and also investigated officially by the National Institute of Standards and Technology (NIST) [12].

The NIST report stated that if there would have been just those fires that were lit because of the kerosene that sprayed around, or only the impact of the airplane (with e.g. almost empty fuel tanks), the towers wouldn't have collapsed. In the actual case, the airplane stripped the insulation (figure 8) from steel structures (that would have prevented the steel itself to heat up), and set fires around the building that made the steel to heat. When heated

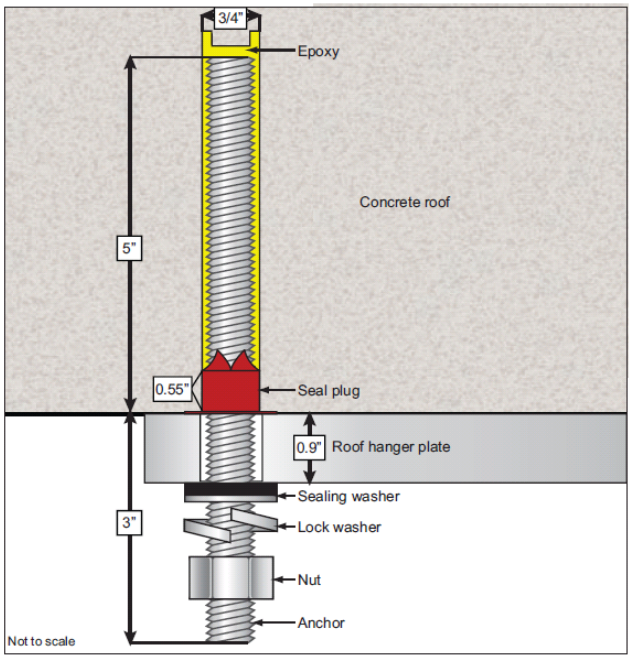

Figure 5: Structure of typical adhesive anchor (the ceiling not in picture) [11]

for long enough time, the bars transferred the load to the other bars, which were not planned to handle so much stress and started to buckle. Amount of this transfer that happened during 100 minutes was in WTC1 10% increase of the load in the north and the south walls, and +25% of the load in the east and the west walls (at the same time the load at the core decreased around 20%). The difference in load increase between the walls was caused by the impact damage of the plane.

Usually in an office fire, the temperature peak can be as high as 1100°C, which is a way below the melting point of structural steel ($T_m \approx 1600$°C). And as it can be seen in simulations (figure 6), steel itself doesn't get as hot, especially when the insulation (figure 8) is OK. But when there isn't insulation, and some supports are missing, more stress is added to the still supporting beams. At the same time the yield strength drops (figure 7), and the creep effect is increased in beams because of increased temperature (temperature effect in creep in figure 24 at page 21) and stress. When creep damage starts to show, beams are more easily buckled, and when some beams

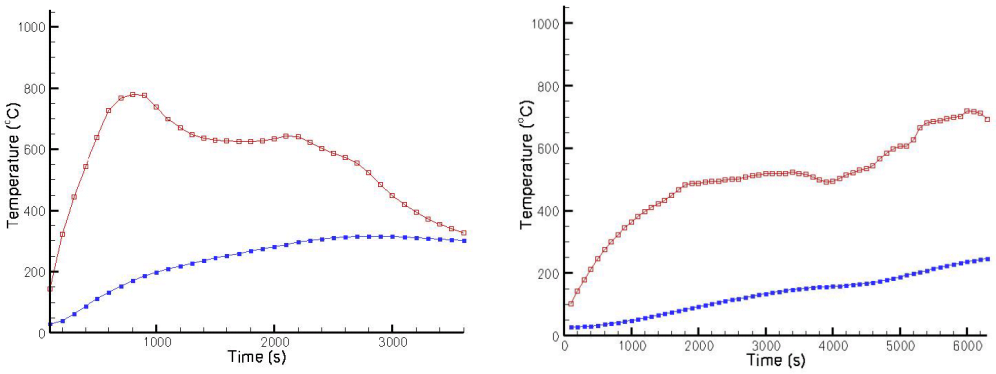

Figure 6: Simulated temperatures of structural steel components in trusses (left) and in perimeter columns (right) [12]

buckle, stress is increased towards others, and damages are occurring at an increasing rate. So creep wasn't the only reason for the collapse, but it was one thing in WTC that made the collapse happen and take as long as it took.

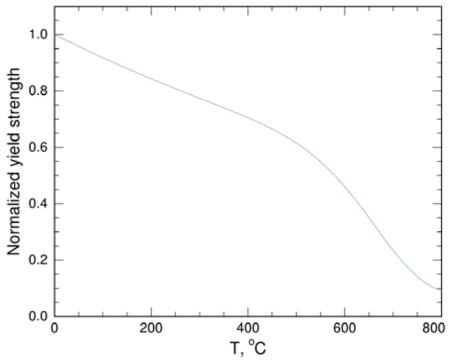

Figure 7: Yield strength temperature dependency of structural steel [12]

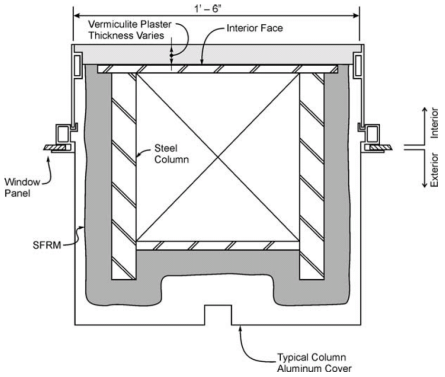

Figure 8: Thermal insulation used in perimeter columns [12]

3.4 Everyday life

In offices, creep can be present every day. Maybe the most commonly known occurrence of creep (without knowing anything about creep) are the paper clips that can't hold paper any more. Plastic paper clips get quite fast un-

usable because of the material. The melting point of plastics is so low that creep effect is present already at room temperature. If plastic paper clips are used to keep a thick pile of paper together and left there for some time, those are not returning to the former stage because of creep relaxation.

Effects of creep can also be seen in old buildings. If there are lead pipes, those can be quite malformed because of lead's low melting point, which makes items made from lead to creep at room temperature (figure 9).

There's also an urban legend that the reason for the thick bottom part of the old windows and unequal surface of old mirrors would have been caused because of the creep. Even though glass does creep, the temperature for it is much higher than that which would be achieved in conventional usage, even with the glass used long time ago [13].

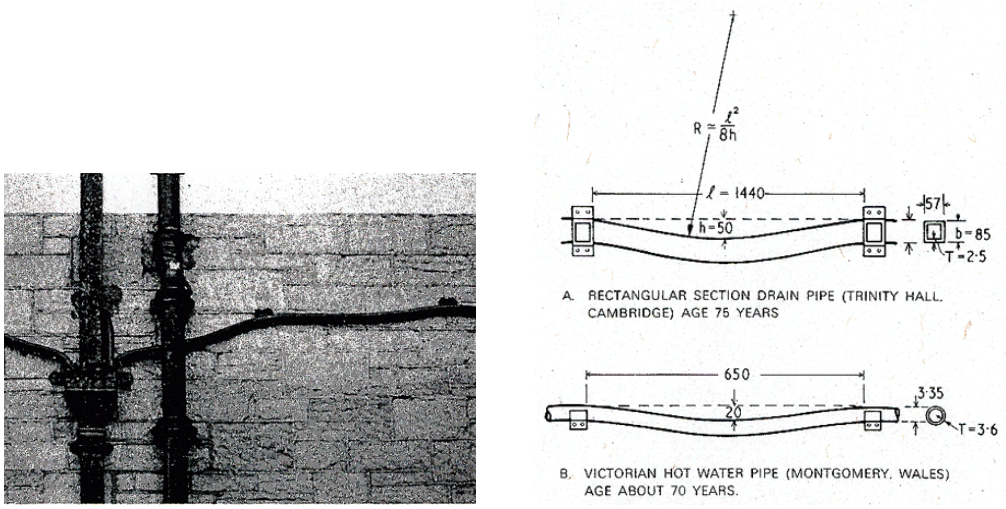

Figure 9: Effect of creep in lead pipes during 70-100 years [14]

Creep is also present in lightbulbs, and can be one cause of those to burn out after a certain time. More common reasons are evaporation from the filament's surface or defects in filament itself, that makes one part weaker and causes it to melt [14]. The temperatures at which filaments operate ($2250 - 2500°C$ in conventional bulbs (25 W and 40 W), and even up to $3160°C$ in photo flood bulbs) are high compared to the regular filament material's, tungsten (symbol W), melting point $3422°C$. If the filament is breaking

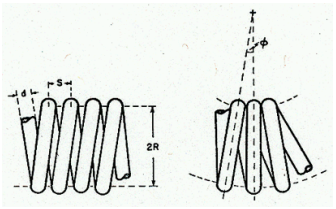

Figure 10: Creep failure of filament caused by torsional creep [14]

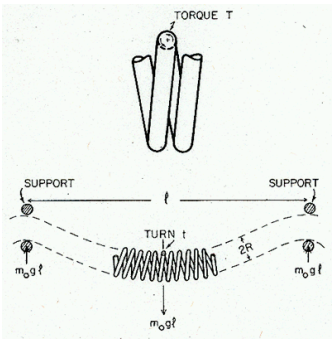

Figure 11: Statistics of horizontal filament [14]

because of creep, it's usually caused by the windings of the filament coil, which may touch each other, causing a short circuit and/or overheating (figures 10 and 11).

If we presume that a filament is defectless, and that it has been made of pure tungsten, it would burn out in about 30 seconds. At the same time, that's the reason, why filaments usually fail because of other reasons; the creep effect is well known, and filaments are really made to be creep resistant by doping tungsten with oxides (e.g. Al_2O_3, SiO_2 and K_2O) [14]. In a sense of the maximum creep rates, the effect of doping is also quite clear; in pure tungsten the maximum creep rate is $10^{-4}\frac{1}{s}$, and with doping only $4 * 10^{-10}\frac{1}{s}$. Effect of doping in deformation mechanism maps can be seen in figures 40 and 41 at page 34.

3.5 Advantages of creep

Although most of the times creep seems to be problematic either for the manufacturing process or for the usage of equipment, there are also some processes that would be quite hard to use without it [15]. These processes (e.g. extrusion ($T \approx 0,6T_m$, $p \approx 35 - 700$ MPa [16]) (figure 12), hot rolling (figure 13), hot pressing and forging) are taken care of at temperatures where the power-law creep (chapter 5.1) is dominant mechanism of deformation. Used pressure can be reduced by increasing the temperature according to equation 10 (at page 26).

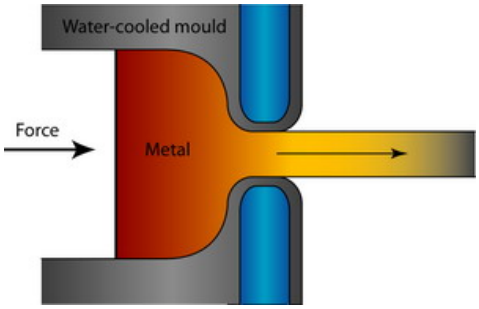

Figure 12: Extrusion process [16]

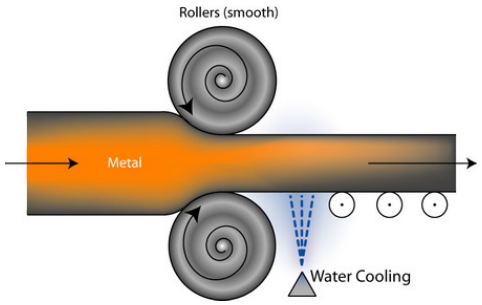

Figure 13: Rolling process [17]

4 Creep as macroscopic phenomenon

4.1 Differences for other metal malformations

Creep isn't the only thing that malforms metals. Within the scope 'leading to rupture without noticeable signs before it happens' there's also fatigue (corrosion leads also to rupture at some point, but is usually visible before that). Difference between fatigue and creep is that in fatigue we're interested about cycles, and in creep about time. Fatigue life is defined as 'number of stress cycles of a specified character that a specimen sustains before failure of a specified nature occurs' by ASTM [18].

Fatigue occurs at the situations, where stress is changing. The definition of creep states that in creep stress and temperature should be constant. E.g. in airplane turbines fatigue is more important, because turbines are started and stopped and the temperature of blades changes quite much. In power plant turbines creep is dominant, because blades are kept at a constant rotation rate at a constant temperature. In real life situations, it might be hard to say which deformation causes the problems, because different deformation mechanisms can also accelerate each other.

4.2 Parameters affecting the creep

There're three parameters that affect always to the significance of creep and can also be seen as parameters in equations; temperature, strain, and material properties. The 'parameters' affecting creep in material are considered in

more closely in chapter 5. A quick overview and terminology are offered in this chapter.

There's some studies about other influences, like radiation [7] and oxidation [19]. These are handled briefly at the end of this chapter.

When studying creep, the temperature used is (usually) so called 'homologous temperature' Θ [14]

$$\Theta = \frac{T_{measured}}{T_{melt}}. \tag{1}$$

Different materials have different temperatures, but the basic rule is that in metals creep occurs only if $\Theta > 0,3$. In ceramics this limit is $0,4-0,5$, and in plastics it is lower than in metals. This is also visible in deformation mechanism maps (chapter 4.3) for different materials. In the maps it can also be seen that at some point (when temperature and pressure are high enough) creep effect is accelerated by dynamic recrystallization (figure 22) [14]. When strain increases, plasticity takes over as the primary malformation mechanism. But it should be noted, that the map shows only the primary mechanism; other mechanisms can be also happening and even helping the primary mechanism (e.g. the reason for power-law creep's strength is diffusion, which can happen either along the boundaries or through the crystal) [15].

Also stress is usually normalized the same way as the temperature, and called a 'normalized shear stress' σ

$$\sigma = \frac{\sigma_s}{\mu}. \tag{2}$$

In this equation σ_s is the applied stress, and μ is the shear modulus [20]

$$\mu = \frac{\tau_{xy}}{\gamma_{xy}} = \frac{\frac{F}{A}}{\frac{\Delta x}{l}} = \frac{Fl}{A\Delta x}, \tag{3}$$

where F is the applied force, A the area the force acts on, and γ_{xy} is the shear strain (see figure 14). Values of shear strain can also be seen in deformation mechanism maps.

In stress, there is also a low limit for creep, but it's not as easy to give some threshold for it as for the homologous temperature. The threshold stress (for

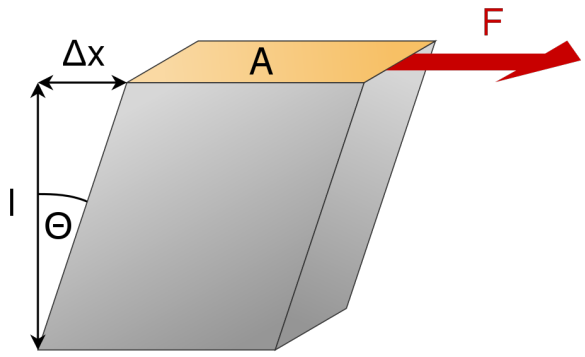

Figure 14: Calculation of shear modulus [21]

creep) is related to the material and it's grain size, but not to temperature . Example showing threshold stress for pure nickel can be seen in figure 15 (more about deformation mechanism maps at chapter 4.3). Usually the threshold is at the bottom of the map and not drawn as a separate line. It should be noted that strain doesn't need to be e.g. added external pressure (gravity is not considered here as an added pressure), but can come from the material itself (e.g. block of material makes usually stress to it's bottom part, or bridges' own weight creates different stress in different parts of it).

As mentioned at the beginning of the chapter, the parameters presented earlier are also visible in the equations, but that doesn't mean that those would be all affecting properties. The atmosphere where the experiments/measurements/usage is made has also influence on the creep effect and it's strength. Oxidation can accelerate the creep process compared to vacuum [19], and even though hydrogen is thought to have only a minor effect on pure copper, it can accelerate creep in doped copper [22]. Atmospheric influence seems to be highly related to the material that's researched/used, and also needs more research generally and per material.

A hydrogen atmosphere isn't directly accelerating creep, but it may be reacting with the oxygen in a material and forming water. This water is formed to microvoids, and because of the temperature of the process, filling those with water vapor of high fugacity. That forms intergranular dimpled fractures mostly at the grain boundaries. This effect is researched with e.g. OFP

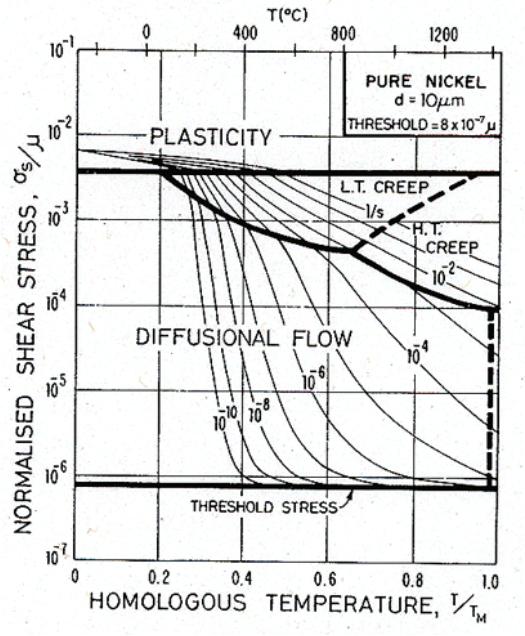

Figure 15: Deformation mechanism map presenting treshold for stress for nickel with grain size of 10μm. [14]

(oxygen-free phosphorus-doped) copper, which had 50 wt. ppm phosphorus. It should be noted, that the material that was researched was 'oxygen-free', i.e. contained only 2 wt. ppm of oxygen. Effect of hydrogen on the strain can be seen in figure 16, and electron microscope image of the damages from the water vapour in figure 17.

Another gas that's researched in relation to the creep process is oxygen and it's absence [19]. Research was made with 2.25Cr-1Mo steel, and the results show that the rupture times were from 1.5 to 3.2 times longer in vacuum than at the same temperature in oxygen atmosphere. This kind of difference is important especially in the 'end-of-life' consideration, because oxidation shortens the time of rupture especially with small objects, which are used in rupture tests. So it would be safe to use thicker objects a longer time than prediction says, which increases costs. The effect is strong especially in ferritic steels, as can be seen from specimen used in figure 18 [19]. The differences between creep rates between air and vacuum in various temperatures can be seen in figure 19.

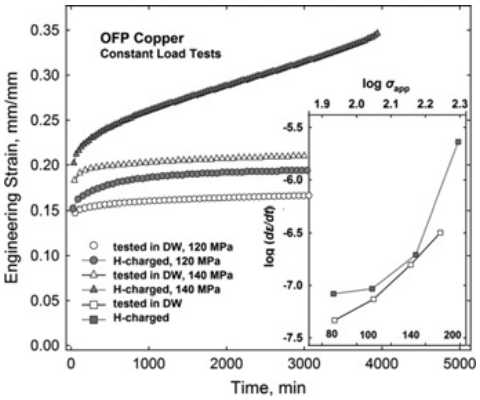

Figure 16: Creep curves with various stresses of OFP copper with and without hydrogen. On the right hand side there's Norton plot showing steady state creep rate vs. applied true stress (from inset) [22]

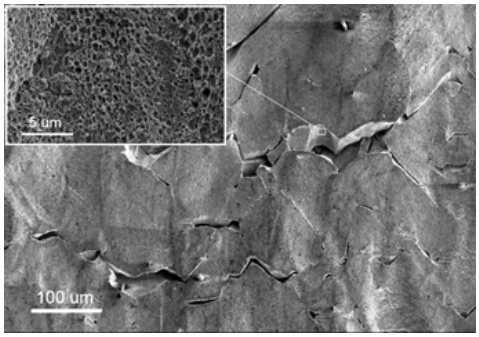

Figure 17: Surface with dimpled facets of OFP copper after being in constant hydrogen charging with temperature of 50°C and strain rate of $10^{-5}s^{-1}$. [22]

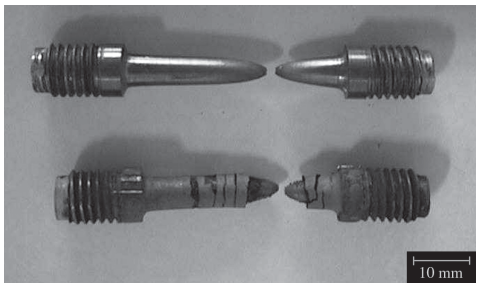

Figure 18: Typical specimens after creep testing at 700°C and 51,7MPa. Top: in vacuum (rupture time $t_r = 162,7$ h), bottom: in air ($t_r = 51,1$ h). [19]

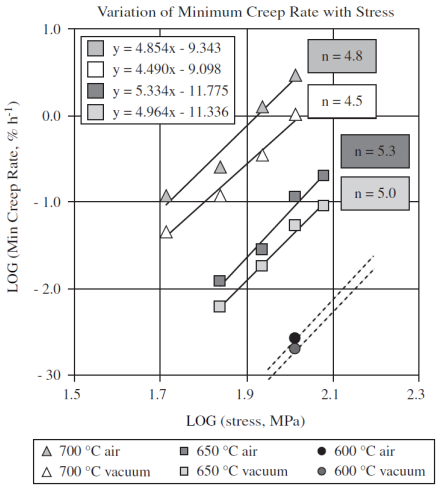

Figure 19: Minimum (= second state) creep rates against stress in various temperatures within air and vacuum. [19]

Also the influence of radiation and flux of high energetic particles has been studied [7]. Quite surprisingly these don't seem to have effect to creep strength.

4.3 Deformation mechanism map

Deformation mechanism map illustrates deformation mechanisms dominance in material within different homologous temperature and normalized stress [14]. Those are often called also with the name of 'Ashby map', after Michael F. Ahsby, who presented those maps first time in the journal *Acta Metallurgica* in 1972 [23]. In this work the most interesting parts of maps are Power-law creep (which contains Low Temperature Creep (L.T. Creep) and High-Temperature Creep (H.T. Creep)) and diffusional flow (areas are named a little different way in different maps). Diffusional flow is also separated in some maps as boundary (meaning Coble creep, chapter 5.2.1) and lattice (meaning Nabarro-Herring creep, chapter 5.2.2) diffusion. Sometimes there is just a vertical dotted line near the melting temperature, which separates the Coble and Nabarro-Herring creeps. Furthermore, when making theoretical calculations about creep, the shear strain rates (see equation 3) from maps could be useful. Shear strain rate is especially good to determine the velocity of creep. These are drawn on maps with thinner black lines.

At the top of the map there's usually an 'ideal shear strength' -line. If stress goes above that, the deformation of perfect crystal ceases to be elastic and changes to catastrophic. It means that the whole crystal structure becomes mechanically unstable. Of course crystal structure can withstand this kind of stress only at 0 K, because otherwise the problem is partly kinetic one; there isn't perfect crystal structure all the time.

Examples for two kinds of maps are in figures 20 and 21. The one in the former figure (20) is the more common way to visualize the map, in which temperature and stress are rising in the positive direction.

Maps are built using various rate-equations for different parts of the map. The most interesting ones for us are those that 'handle' parts where creep is

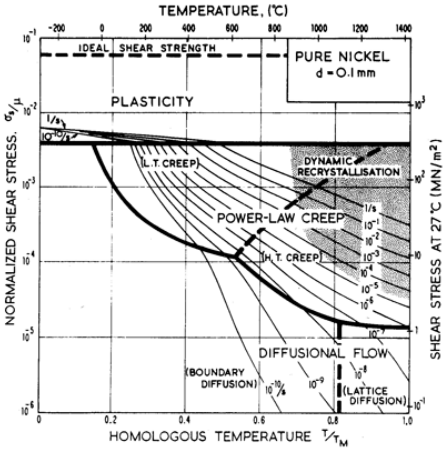

Figure 20: Deformation mechanism map of nickel with grain size of 0.1 mm. [14]

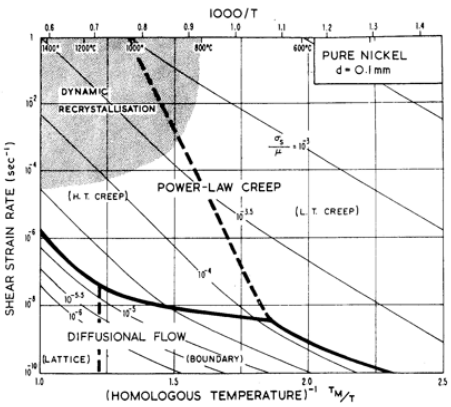

Figure 21: Deformation mechanism map of nickel with grain size of 0.1 mm using reciprocal temperature. [14]

dominant. These are rate equations for power law creep (chapter 5.1):

$$\dot{\gamma}_4 = A_2 \frac{D_{eff}\mu b}{kT}\left(\frac{\sigma_s}{\mu}\right)^n, \tag{4}$$

the rate-equation for Harper-Dorn creep (chapter 5.4)

$$\dot{\gamma}_5 = A_{HD} \frac{D_v \mu b}{kT}\left(\frac{\sigma_s}{\mu}\right), \tag{5}$$

the rate-equation for power-law creep and power-law breakdown:

$$\dot{\gamma}_6 = A_2' \frac{D_{eff}\mu b}{kT}\left[\sinh\left(\alpha' \frac{\sigma_s}{\mu}\right)\right]^{n'} \tag{6}$$

and the rate-equation for diffusional-flow (chapter 5.2)

$$\dot{\gamma}_7 = \frac{42\sigma_s \Omega}{kTd^2} D_{eff}. \tag{7}$$

It can be seen, that Harped-Dorn creep (equation 5) is 'just' 1-power-law creep. $A_{HD} = \rho_m \Omega b$ (where ρ_m is the density of mobile dislocations and Ω

atomic or ionic volume), and it has experimental values for some materials. This is discussed further chapter 5.4.

It should be noted, that equation 4 contains a model for both low and high temperature creep. In the high temperature low stress part the lattice diffusion dominates (H.T. creep), and at the low temperature and/or high stress, the core diffusion dominates (L.T. creep). This also affects the strain rate variation; in the H.T. area it varies as σ_s^n, and in the L.T. as σ_s^{n+2}.

In all rate-equations, D_{eff} is the effective diffusion coefficient (different in each equation, depends on creep mechanism and material), k is Boltzmann's constant, σ_s applied stress, T is absolute temperature, μ shear modulus and b the magnitude of the Burgers vector. In the diffusional flow's rate equation, d is the grain size (which isn't explicitly affecting the other presented rate-equations). A_2 in equation 4 is just a dimensionless (material dependent) constant that can go up to 10^{15}. It's relation to the constant A (Dorn constant) is $A_2 = \left(\sqrt{3}\right)^{n+1} A$, and the values of A can be obtained from various tables (some listed in Deformation-Mechanism Maps -book [14]).

In maps there's also a grey area stating 'Dynamic recrystallization' (see figure 22), which makes things a bit trickier. In that area, old grains disappear and new ones form. When this happens in a way that a 'creeped' grain is replaced by an 'uncreeped' one, creep rate increases because creep can start again from primary creep. Usually dynamic recrystallization needs the temperature be quite high, over $0,6T_m$.

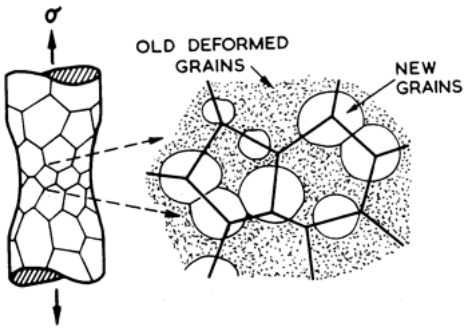

Figure 22: In dynamic recrystallization deformed grains are replaces by new undeformed once, which can be affected by a new creep process starting from primary creep. [14]

A warning about maps; all deformation mechanics maps should be considered to be approximations, and the values not read too closely. This doesn't mean that maps would be wrong, but they're only as good (or as bad) as it's possible to make with current knowledge (meaning equations and data used to form those). But if the homologous temperature and normalized shear stress point to the middle of some processes area, then it's quite clear that the process is at least dominant. Even then, it's not necessarily the only affecting process.

4.4 Phases of creep

4.4.1 Primary (transient) creep

Primary creep is the first deformation that happens to matter when heat and strain (e.g. pressure) are added. It 'hardens' the material. Length of the primary creep stage is quite short, and the time of stage isn't changed much when the temperature is raised. When temperature is risen, the strain in material is of course higher, but material moves to secondary stage creep always after the same time (as can be seen also in figure 24).

At microscopical level of this stage, grains slide to positions in which they're 'easy' to move. Grains kind of arrange to the best possible formation without need to malform. Meanwhile, more and more dislocations are formed, which start at some point to prevent the creation of new dislocations.

From as metallurgical point of view, during the first phase there's mostly occurring hardening at the beginning, but the rate of annealing increases with time. When the rate of annealing and hardening gets equal, the process is advanced to the secondary creep. As mentioned earlier, dynamic recrystallisation may make things trickier, and may cause the whole bulk material not to be at the same creep stage simultaneously.

Also during the primary creep, deformation is partly elastic, so grains can also return to original places if pressure is removed. If strain is small enough, all deformations are elastic. Then there is a lower limit for which stress/strain is needed for creep. This can be seen also in the example creep curve in figure 25 at page 22, where the elastic area is the same as the one marked with

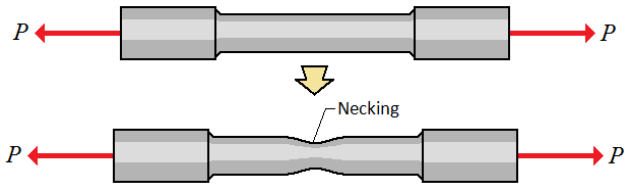

Figure 23: Necking in bulk material [25]

'Instantaneous deformation' (which happens only if strain is high enough to start creep). Other example is at deformation mechanism map presented in figure 15 at page 14.

4.4.2 Secondary creep (steady state creep)

In secondary creep hardening and annealing (thermal softening) are in balance, which makes creep to proceed at a constant rate. The strain rate increase ($\dot{\epsilon}$) is also at the minimum. An equation for the steady state creep for most of the creep types is equation 10 at page 26.

Importance of secondary creep rate comes from engineering; components should never move from secondary state to tertiary. This means that most components working at temperature and stress where creep is applicable should be designed to stay as long as possible in the secondary creep stage. More about testing is in chapter 4.5.

There isn't any visible changes during the secondary creep. In the microscopical level amount of dislocations isn't changing, even though they are moving.

4.4.3 Tertiary creep

Tertiary creep leads to rupture, and it happens usually in quite short timespan when material moves out from secondary creep state [24]. Tertiary stage is also the first (and only) one showing some damages in material; before it those upcoming defects are invisible. Increasing rate of creep strain (and short time of whole tertiary stage) is caused by necking (see figure 23).

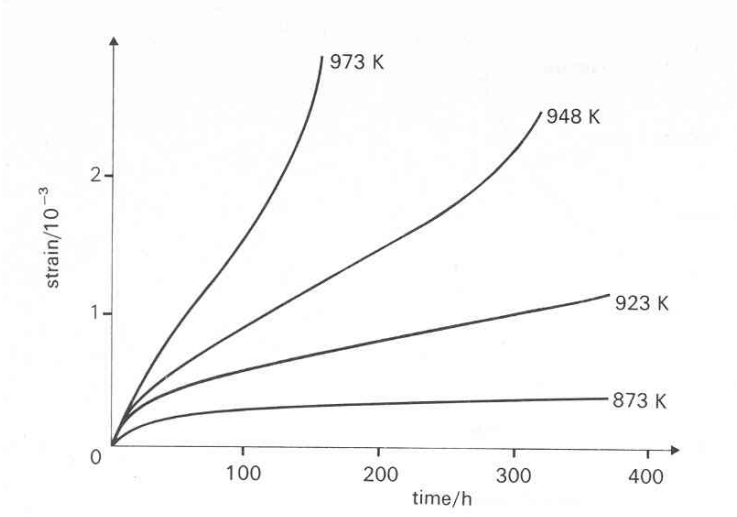

Figure 24: Example of low alloy steel creep curves at 70 MNm^2 stress. [26]

4.5 Testing and measuring creep

There's standard test set E139 [28] for creep by ASTM, whose the whole name is 'ASTM E139 - 11 Standard Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials'. Tests can be used for both engineering and research needs for materials. The difference is that in engineering tests, there are usually so called 'stress-rupture' or 'creep-rupture' -tests, where a heavy load is used (because of interest to time of rupture or service life), while in research tests are more often 'creep' tests. In a creep test, the time of the test and the stress are way below those that cause rupture in materials, because the goal is to try to understand creep in a more detailed way.

The test itself is quite simple; a specimen is put in furnace set at constant temperature, and constant stress is added to the specimen. Then the dimensional changes of the specimen are measured with the time, if making either creep or creep-rupture test. Time depends on which test is made; at the stress- and creep-rupture measurements are done until the specimen breaks, and in creep tests, just a predefined, usually quite long, time. In a stress-rupture test the only interesting thing is the time that specimen needs to rupture, so

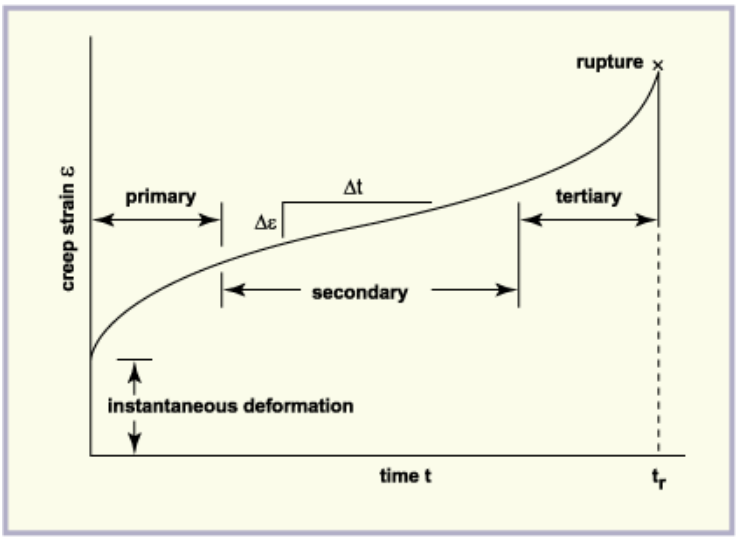

Figure 25: Example of creep curve, where pressure/stress is kept constant (and over the limit of elastic deformation) [27]

no other measurements are needed. One kind of test equipment can be seen in figure 26.

The creep and creep-rupture tests are harder to make than the stress-rupture test. Former requires stable temperature (fluctuation can be maximum $0,1-0,5°C$). Also the measurements need to be done in precise way, e.g. in materials with low creep, the displacement resolution should be around 5 nm. In addition, it is important to keep the added stress uniaxial and uniform towards the specimen. In a stress-rupture test, the interest isn't to know e.g. how big deformations are; just to figure out the time when the specimen breaks.

The result of all tests is a graph similar to the one in figure 25 at page 22.

4.6 Effects of creep

4.6.1 Creep relaxation

Creep relaxation is important especially in relaxation-limited applications (e.g. hot turbine casings and steam pipes which both have a high temperature

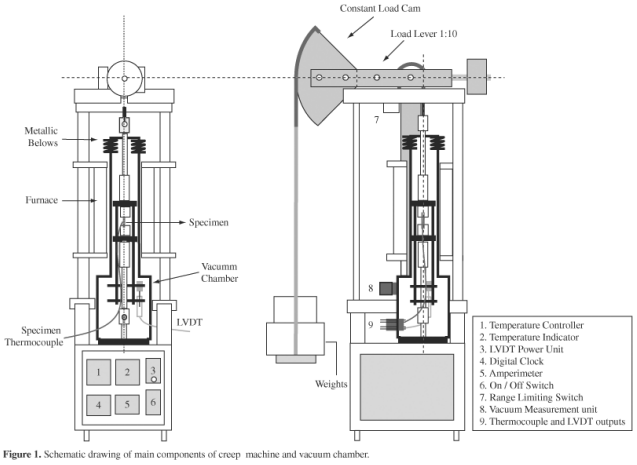

Figure 26: Setup for creep testing with possibility to conduct tests also in vacuum [19]

at least at the other side). In these cases the important question might be how often bolts should be tightened that steam won't leak.

Bolts get loosened because of creep relaxation, which makes creep strain to replace elastic strain. So, the length of the bolt, which keeps the pipes together, stays same, but the elasticity that pulled the bolt's head and nut towards each other is lost. This effect is also the one that causes the paper clips to loose the grip. It's presented graphically in figure 27.

Because the total length of the bolt needs to stay constant, also total strain ϵ_{tot} should be constant [15], which means that at any time t

$$\epsilon_{tot} = \epsilon_{elastic} + \epsilon_{creep}, \tag{8}$$

where $\epsilon_{elastic} = \frac{\sigma}{E}$ and $\epsilon_{creep} = B\sigma^n$ (latter when temperature is considered as constant). Because ϵ_{tot} is constant, we can differentiate equation 8 and then integrate it from $\sigma = \sigma_i$ (σ_i initial strain) at $t = 0$ to $\sigma = \sigma$ at $t = t$, which gives

$$\frac{1}{\sigma^{n-1}} - \frac{1}{\sigma_i^{n-1}} = (n-1)BEt. \tag{9}$$

With equation 9 it's possible to calculate the interval for retightening after deciding how much the strain σ is allowed to drop from it's original value (and

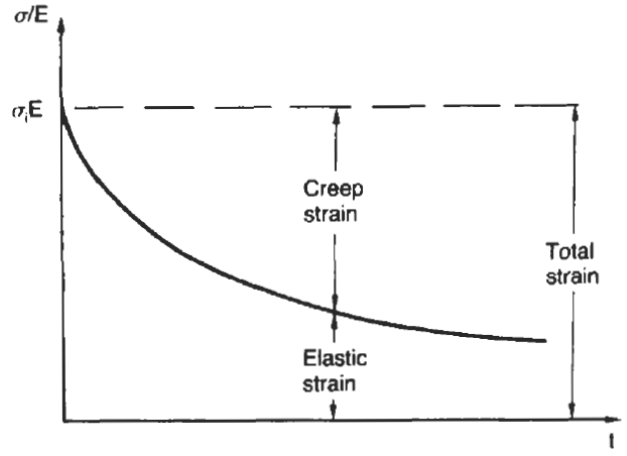

Figure 27: Elastic strain replacement by creep strain at a high temperature [15]

of course knowing the characteristics of the material of the bolt). It's worse to try to cope with the relaxation by tightening bolts even more; that just increases the strain and the rate of this relaxation process.

4.6.2 Creep damage

In 'long-life creep situations', e.g. turbine blades (in military aircrafts) the more interesting variable is the steady-state creep rate $\dot{\epsilon}_S$; this can be obtained from creep curve's secondary part's slope ($\frac{\Delta\epsilon}{\Delta t}$) (see picture 25). In these situations it's important that no malformations, which would change the dimensions of target, occur. As a design perspective these are called either 'displacement-limited' (e.g. turbine blades) or 'buckling-limited' (e.g. aircraft wings) designs.

Creep damage is visible only in the tertiary creep stage, when cavities start to form. That causes necking, which puts the same stress as previously into a smaller area/volume of material, increasing the creep rate. Because the tertiary stage of creep is usually short, in displacement limited applications, the end of secondary creep is the most interesting information.

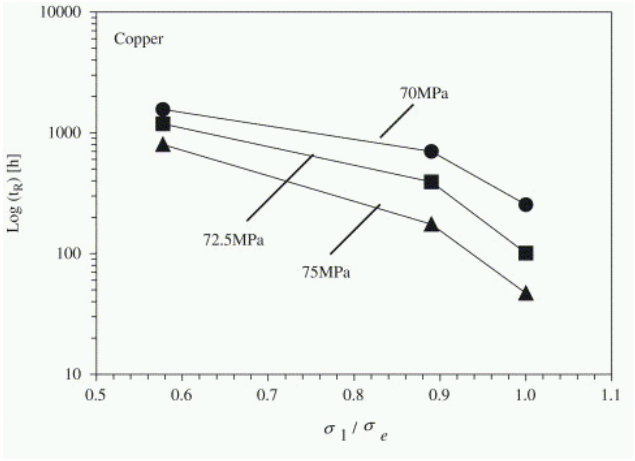

Figure 28: Creep-rupture diagram for copper with various stresses [29]

4.6.3 Creep fracture

Creep also needs to be known when machines are built to be used at high temperatures and/or high pressures. The value that designers are interested in is usually time-to-failure (also called time-to-rupture and rupture lifetime) ($t_f = A'\sigma^{-m}\exp^{+(Q/\overline{R}T)}$, where A',m, and Q are creep failure constants, not creep constants [15]); i.e. how long it's safe to use a given machine. These are also called 'short-life creep situations' or 'failure-limited design'. This is important, e.g. for the casing on a rocket combustion chamber or pressure pipes. In these cases also the malformation is acceptable as long as there isn't failure.

Time-to-failure results are usually presented in creep-rupture diagrams (figure 28), where either temperature or stress are varied to show the effect of environmental changes for lifetime.

5 Physical mechanisms of creep in metals and ceramics

All creep mechanisms follow the Arrhenius's law, and are usually limited by the rate of the diffusion.

Mechanism	m	q	Chapter
Diffusional flow (Nabarro-Herring creep)	1	2	5.2.2
Diffusional flow (Coble creep)	1	3	5.2.1
Grain boundary sliding through crystal lattice	2	2	5.3
Grain boundary sliding along grain boundaries	2	3	5.3
Dislocation creep	3-8	0	5.1

Table 1: Creep exponents in Dorn equation (equation 10) for various physical mechanisms [30]

Arrhenius's equation can be written in general form as

$$k = Ae^{\frac{-E_a}{k_B T}},$$

where k is a rate constant, T absolute temperature, A a pre-exponential factor (just constant for the current situation), E_a activation energy and k_B Boltzmann's constant. In some situations (e.g. in creep) Boltzmann's constant is usually replaced by gas constant R.

In creep the equation for steady state creep rate $\dot{\epsilon}_{ss}$ can be written in a form, which is the so called Dorn equation [30]:

$$\dot{\epsilon}_{ss} = Ae^{\frac{-Q}{RT}} = A'\sigma^n e^{\frac{-Q}{RT}} = \frac{A_2\sigma^m}{d^q T} e^{\frac{-Q}{RT}}, \tag{10}$$

where σ is the applied stress, d the average grain diameter, R the universal gas constant and Q activation energy (for self-diffusion, when $T \geq 0,5T_m$). The values of m and q depend on the creep mechanism, and values for different processes can be found in table 1. This equation works best for diffusional flow -situations, and isn't describing e.g. Harper-Dorn creep (chapter 5.4). There's also a generalized Dorn equation, which applies to all creep mechanisms:

$$\dot{\epsilon}_{ss} = \frac{A(D_0 e^{\frac{-Q}{kT}} Gb}{kt} \left(\frac{\sigma}{G}\right)^n \left(\frac{b}{d}\right)^p, \tag{11}$$

where D_0 is diffusion coefficient at infinitely high temperature, G is the shear modulus, n is a stress exponent and b the Burgers vector

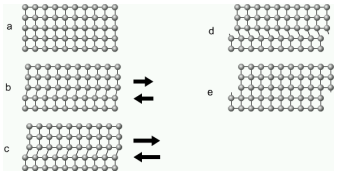

Figure 29: Dislocation moving in a perfect crystal structure [31]

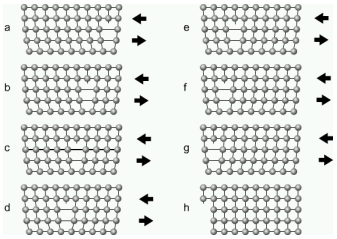

Figure 30: Dislocation moving through a crystal [31]

Total strain ϵ can be calculated with the Garofalo's relationship:

$$\epsilon = \epsilon_0 + \epsilon_t \left(1 - e^{-rt}\right) + \dot{\epsilon}_{ss} t, \tag{12}$$

where ϵ_0 is instantaneous strain (before primary creep begins), ϵ_t the limit for primary creep (meaning the strain when primary creep changes to secondary creep), r is the ratio between creep rate and -strain, and ϵ_{ss} is the secondary creep rate.

5.1 Dislocation creep

Dislocations produce power-law creep (also called n-power law creep and driven creep), in which increasing the stress increases the creep rate with the exponent of $3 - 8$ (m in Dorn's equation (10)). A normal dislocation needs quite high stress added to a crystal structure that dislocations start to move. In a creep situation, stress needed for dislocations to move is usually lower, because diffusional creep 'unlocks' obstacles that prevent dislocations to move. Of course, if stress is high enough, dislocations can climb over the obstacles.

The reason for n-power law creeps' strong dependency on applied stress comes from this climbing; when more stress is applied, the climb force is greater. With a higher climb force, more dislocations climb over precipitates, and are allowed to glide more and to increase the strain rate.

If the stress rises high enough, creep rate starts to increase even faster than the theory of power-law creep suggests. Area with higher stresses than power-

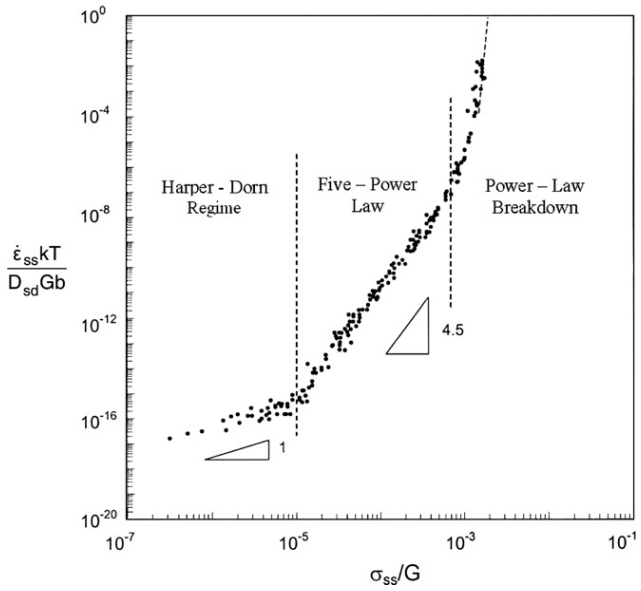

Figure 31: Compensated steady-state strain rates versus modulus compensated steady state stress for pure aluminium [32]

law creep predicts is called 'power-law breakdown'. At that area, the relation between stress and creep rate isn't linear anymore, but starts to increase in an exponential way. In deformation mechanism maps, that area is marked as dislocation or plasticity. In that part the mechanisms that's usually happening is dislocation glide, where the potential wall needed for dislocations to move is overcome with thermal activation and great stress. Dislocation glide and dislocation creep are different phenomena, and shouldn't be confused. The former makes plasticity, the latter power law creep.

5.2 Diffusional creep

Diffusion produces linear-viscous creep (meaning 1-power law creep, $\frac{\text{Strain rate}}{\text{Stress}} = 1$). Usually both Coble and Nabarro-Herring creep are occurring in parallel, and not completely separated processes. In both atoms are moving because of the diffusion in the grain from one place to another. There's just difference of the 'route' the atoms use (see figure 32). The diffusional creep rate is proportional to the applied stress σ (as can be seen also from rate-equation

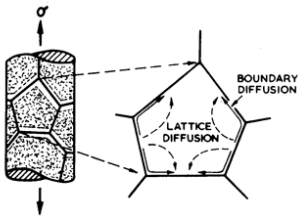

Figure 32: Movement of atoms in Coble (Boundary diffusion) and Nabarro-Herring (Lattice-diffusion) creeps [14]

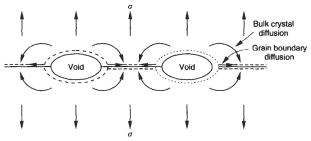

Figure 33: Formation of voids because of diffusional flow at grain boundaries [15]

7). Diffusional creep usually happens with grain boundary gliding, which prevents formation of holes between grains (as can be seen in figure 34).

5.2.1 Coble creep

Coble creep is named after Robert L. Coble, who reported his theory 1963 in Journal of Applied Physics [33]. In Coble creep vacancies (point defects) move along grain boundaries or, if the material is a single crystal, at surfaces (figure 32). Coble creep occurs at lower temperatures, than Nabarro-Herring, because the activation energy Q is smaller for grain boundary diffusion than for self diffusion. Usually Coble creep is dominant when homologous temperature $\theta < 0.7$.

The rate of Coble creep scales with respect to grain size as $\frac{D_b}{d^3}$. The reason for a quite rapid drop the creep rate is because with larger grains, there's fewer boundaries. The number of boundaries also limits the temperature dependency of Coble creep. Temperature increase doesn't increase the rate as much as in Nabarro-Herring creep, because of the limited number of nearest neighbours at the boundary.

5.2.2 Nabarro-Herring creep

Nabarro-Herring creep is named after Frank Reginald Nunes Nabarro and Conyers Herring [34]. Nabarro released the report containing the idea of creep nowadays called Nabarro-Herring creep in 1948 [35]. Herring published

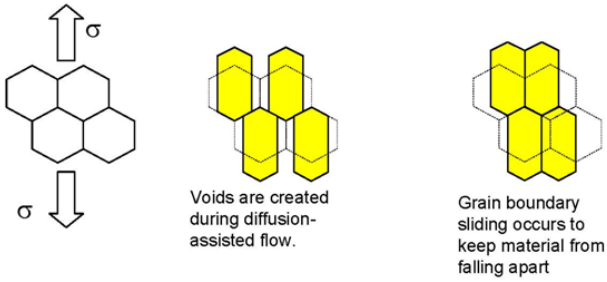

Figure 34: Mechanism of grain boundary sliding [30]

his paper containing more elaborate version of same effect in 1950 [36]. In Nabarro-Herring creep, vacancies (point defects) move through a crystal/grain (figure 32) towards a stress axis, causing at the same time the grain elongation. It needs higher temperature then Coble creep in order to be the primary process of creep (because of higher activation energy Q for self diffusion). Usually Nabarro-Herring creep is dominant when the homologous temperature $\theta > 0.7$. Size increase in the grains doesn't have as strong influence on the rate than in Coble creep, and the creep rate scales as $\frac{D_v}{d^2}$.

5.3 Grain boundary sliding

Usually grain boundary sliding occurs with diffusion creep, when voids are formed between grains, and those voids are filled by grains moving back together. In sliding the grain boundary the grain elongation shouldn't be happening, as they just move in current form to fill the gaps between (as seen in figure 34). Simultaneously the number of grains along the tensile axis increases [37]. This type of behaviour is generally called Rachinger sliding.

Grain boundary sliding can result in superplasticity, which allows materials just stretch without breaking. This happens only if grain size is small (typically $d \leq 10\mu$m). At that time sliding happens through crystal lattice ($m = 2$ and $q = 2$ from table 1 to equation 10).

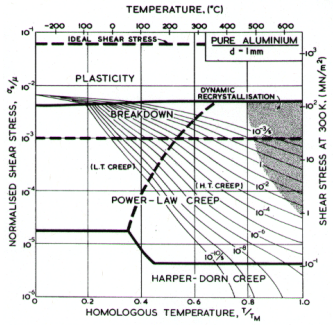

Figure 35: Deformation mechanism map for big grained aluminium showing Harper-Dorn creep [14]

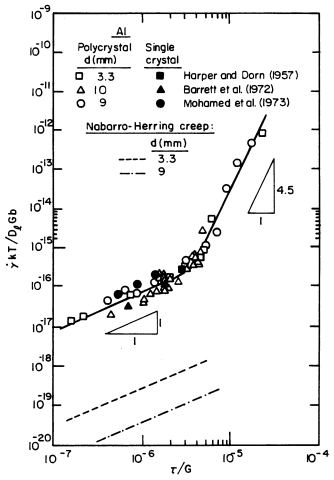

Figure 36: Experimental data of pure Al supporting the occurence of Harper-Dorn creep at low stresses. Theoretical prediction for Nabarro-Herring diffusion creep is added with broken lines [37]

5.4 Harper-Dorn creep

Harper-Dorn creep is a mechanism usually suggested to occur in various materials at low stresses (as pictured in figure 35). Suggested, because even though it was published first time at 1957 [38] with big grained aluminium, there has been debate ever since if this kind of creep mechanism really exists. Some papers say that it's reproducible, some that it isn't, and some state that those who can't reproduce the results are either using wrong kind of material and/or too high pressure. Harper-Dorn creep occurs only when stress $\sigma < 0,1$ MPa [39].

The original results about occurrence of Harper-Dorn creep were noticed with big grained aluminium. Later researches have also found other materials that have shown effects interpreted as Harper-Dorn creep, but as mentioned, there is still discussion about the correctness of results.

The mechanism for why Harper-Dorn creep exists or what microscopical events are creating it are not clear. It has been seen that at low stresses there's

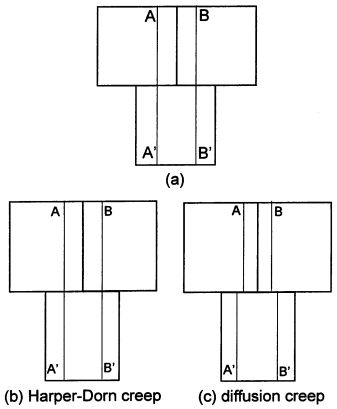

Figure 37: (a) An array of three grains with longitudinal marker lines AA' and BB' showing the effect of (b) Harper-Dorn creep and (c) diffusion creep. [37]

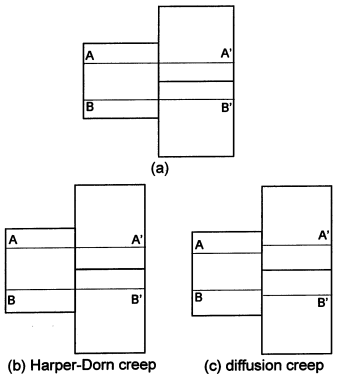

Figure 38: (a) An array of three grains with transverse marker lines AA' and BB' showing the effect of (b) Harper-Dorn creep and (c) diffusion creep. [37]

creep that has almost 1400 times higher rate than theoretical suggestions of Nabarro-Herring and Coble creep (figure 36).

The things that separate Harper-Dorn from Nabarro-Herring creep and grain boundary sliding are that in Harper-Dorn marker lines stay at the same place when grains elongate (figures 37 and 38), and the value of sliding contribution ξ is low

$$\xi = \frac{\epsilon_{gb}}{\epsilon_t}, \tag{13}$$

where ϵ_{gb} is the strain due to grain boundary sliding, and ϵ_t total strain in sample. These things support the hypothesis, that Harper-Dorn creep is a unique creep mechanism.

6 Increasing creep resistance

When designing materials to be used (or manufactured) in a creep-friendly environment, there's multiple ways to try to cope with creep effects. Maybe the easiest one is to try to change temperature and strain to a range that's not suitable for creep. In turbines the temperature can also be decreased by introducing hollow channels / cooling ports in blades, which let air flow

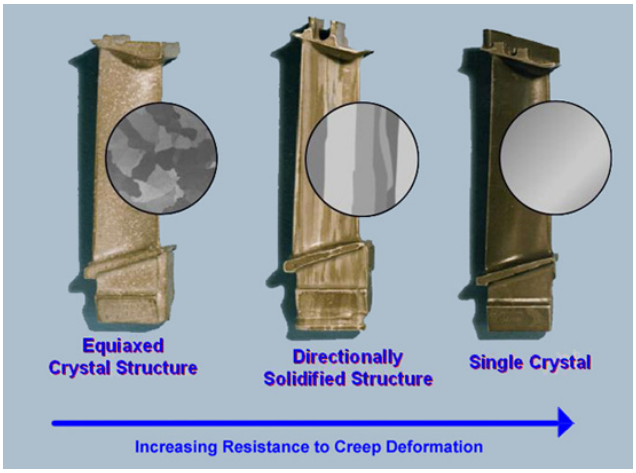

Figure 39: Various crustal structures and and creep resistance [15]

through and keeps blades cooler. This kind of cooling also makes it possible to have a heat engine that works with higher temperature than the melting point of blade material. But usually decreasing the temperature and pressure are not suitable candidates because of e.g. simultaneously dropping energy output (in heat engines).

Other quite often used possibility to modify the grains by either changing their size or arranging them to a better formation. Former can be done by increasing grain size, which decreases the diffusion effect (diffusion rate scales as $\frac{D}{d^n}$, where n is 2 for Nabarro-Herring creep and 3 for Coble creep). Even though diffusional flow decreases as the grain size increases, it doesn't disappear. Also when grains are big enough and stresses are low, Harper-Dorn creep needs to be taken into consideration.

There's a possibility to 'arrange' grains to a more creep resistant form. This is used e.g. in turbine blades by moulding blades which contain long parallel grains instead of random grain locations. Long grains have very large diffusional distances, and have no driving force for grain boundary sliding or cavitations in grain boundaries. These long grains should also be arranged in a way that grain boundaries are parallel to stress. This kind of an alloy is called 'Directionally solidified' (DS) alloy (figure 39).

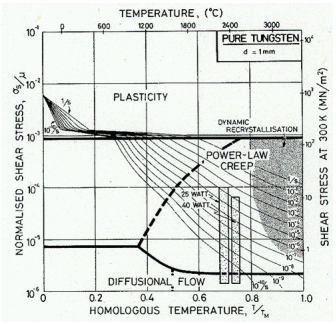

Figure 40: Deformation mechanism map for pure tungsten with grain size of 1 mm. Shaded box show normal operating area for normal light bulbs [14]

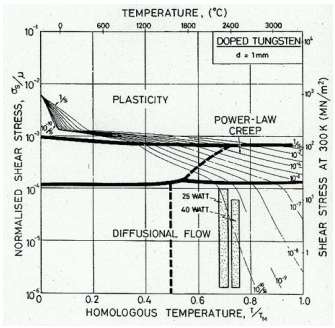

Figure 41: Deformation mechanism map for doped tungsten with grain size of 1 mm. Shaded box show normal operating area for normal light bulbs [14]

A third possibility is to dope a material with non-deforming particles, usually oxides. These particles are able to inhibit the recrystallization (which increases the creep rate), and also keep the grain boundaries together. This method is used in so called 'super alloys', e.g. in turbine blades, and can contain even around 10 different materials [15]. When e.g. tungsten is doped, the grains are also elongated, which further prevents creep effects. Examples of doping effect can be seen in figures 40 and 41, and the effect to usage time in chapter 3.4. Maps presented in figures can be used to check the requirements for the materials; if temperature rises, stress needs to be lowered to obtain same lifetimes, or accept the shortening of lifetime. In bulbs lowering the stress could be made e.g. by adding more supports for filament (filament statistics in figure 11).

In a way one possibility is just to change material itself to something more creep resistant. In metals, the super-alloys are at the moment top-of-the-line choices, but e.g. ceramics are even better; they are lighter, have higher melting temperature, and that way lower the creep. Problems include low thermal conductivity (in some ceramics) and especially very low toughness.

7 Current research in creep

7.1 Overview

Even though most of the references are quite old (especially books about creep and deformation mechanism maps), there's around 60'000 publications in the Web of Science containing creep in the topic. Only little over 1400 have something to say about creep and copper. In many references it was mentioned that aluminium is the most studied material, and it is seen also in Web of Science; there was almost 3000 publications about creep and aluminium. One reason might be super-alloys, where aluminium is usually used. At the same time, research about creep in steel has produced over 10'000 publications.

Because of the works initial reason was accelerator structure making for CLIC, even more interesting research than research of creep in copper is research of creep in OFHC copper. And that had been done also, as can be seen in figure 43.

14 papers containing creep, OFHC and copper in topics have been published between 1958 and 2011. 9 of those has been published after the year 2000.

As can be seen from figures 42 and 43, research about copper isn't following the 'trend' in creep research. Especially publications between 2005-2009 have much lower ratio compared to all creep publications.

7.2 Closer look to CLIC related articles

Because of the initial interest from making accelerator structures for CLIC, I collected some papers that are related to the methods used when building the structures or the problems that have occurred during the manufacturing process.

Maybe the most interesting paper is 'Tensile creep behaviour of coarse grained copper foils at high homologous temperatures and low stresses' [40], which was published in 2005. This work describes testing of creep effects with very high homologous temperature (θ in range $0,83 - 0,99$) and low stresses ($0,1 - 0,6$ MPa). The material researched was OFHC copped foils

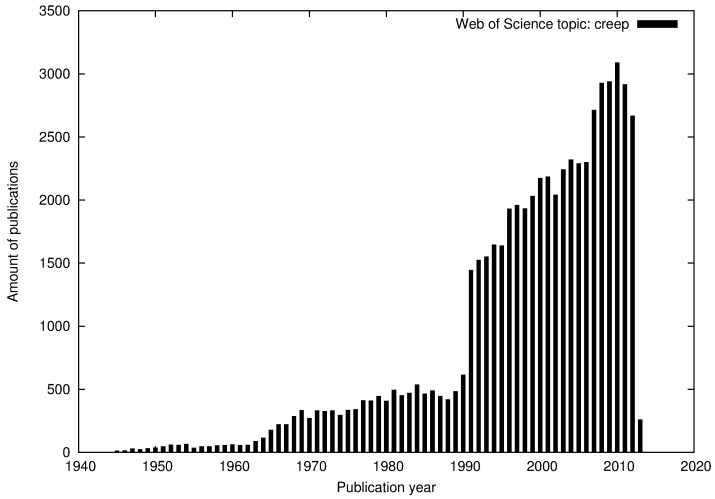

Figure 42: Publication about creep in Web of Science. Total count of publications 59683

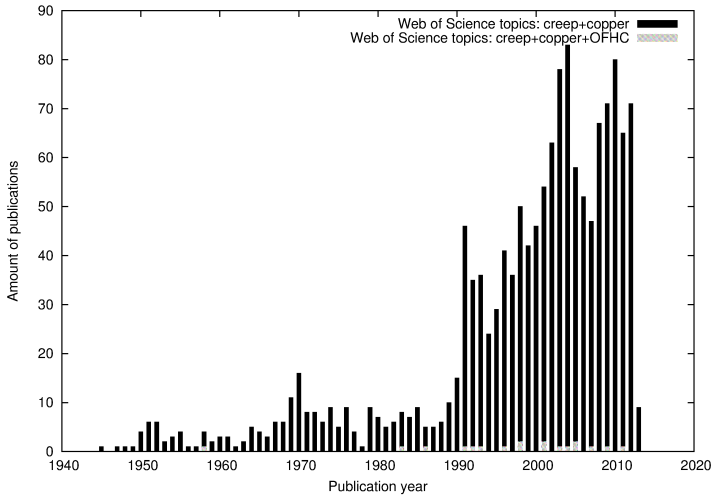

Figure 43: Publications about creep with copper and OFHC copper in Web of Science

(thickness of 0.4 mm and 0.6 mm) with a grain size about one mm so it had grain size larger than in CLIC, which had average grain diameter of $65\mu m$. Interestingly there was no indication of Harper-Dorn creep, but something else with a stress exponent of 2 (In Harper-Dorn creep it should be 1). Research made in that paper could be applied at least to the discs at the top of the disc stack. Problem with lower ones is the pressure difference because of gravity.

OFHC copper is researched more generally in 'Kinetics of high-temperature deformation of polycrystalline OFHC copper and the role of dislocation core diffusion' [41]. The mechanism driving the rate of creep was suggested to be dislocation core-diffusion (dislocations helped by Nabarro-Herring creep). As mentioned in chapter 6, one way to prevent the creep effect is to add oxides to keep grains together and prevent diffusion. With oxygen content of about 40 ppm, the oxides were not preventing the diffusion. At high temperature range ($750 - 950°C$) the main process of creep was suggested to be plastic flow controlled by grain boundary diffusion (Coble creep).

Copper's creep effects have also been researched with atomic force microscope in 'Microstructural evidence for diffusional creep in copper using atomic force microscopy' [42]. That research shows that there doesn't seem to be Harper-Dorn effect at least when examining grain sizes in OFHC copper. Problem with the statement is that the used stresses (σ from 1.6 to 8 MPa) are much higher than suggested to be suitable for Harper-Dorn creep ($\sigma < 0,1$ MPa, chapter 5.4). Too big a stress has been said to be invalidating for quite many of the papers stating Harper-Dorn creep doesn't exist.

8 Summary

Even though creep is known from the 19th century and the first publication has been made at 1910, it still involves several unclear things. It's quite clear, that creep happens in three stages, and that the driving mechanisms for creep are both diffusion and dislocation. It's clear that diffusing vacancies can move along boundaries (Coble creep) or through the crystal (Nabarro-Herring creep). It's also clear that dislocation produces n-power-law creep, which increases the rate of creep with exponential dependency of strain. And

it's known that creep can occur in all solids, and needs always temperature that's 'high' compared to the materials melting point.

What's unclear is e.g. creep effects at lower stresses; does Harper-Dorn creep exists, and if it does, what's the mechanism that makes it? When new materials are discovered and produced for high temperature applications, there's always new questions about creep resistance of the materials.

Even without the new unclear things and materials, there's still research to do even with 'old' materials despite of over 100 years of research about topic. And when making super-alloys, creep is the thing that needs to be kept in mind all the time.

In theory preventing the creep is easy, but in practise creep resistance is almost all the time a compromise with some other parameter (e.g. weight, hardness, fragility, cost).

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